Properties and Improved Space Survivability of POSS Polyimides



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Report Documentation Page

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Outline



- > Lower Earth Orbit Environment
- Assessment of state-of-the-art Space Polyimides.
- ➤ POSS: Polyhedral Oligomeric Silsesquioxane
- ➤ POSS Kapton Polyimides
- ➤ Ground Based Tests: Atomic Oxygen (AO) Erosion Studies
- ➤ Self Forming / Self Healing Silica Passivation Layer
- > Modeling and Simulation of AO attack on POSS
- > Flight Tests: MISSE 4, 5, 6
- > Thermal and Mechanical Properties
- > Summary and Conclusions
- > Acknowledgments



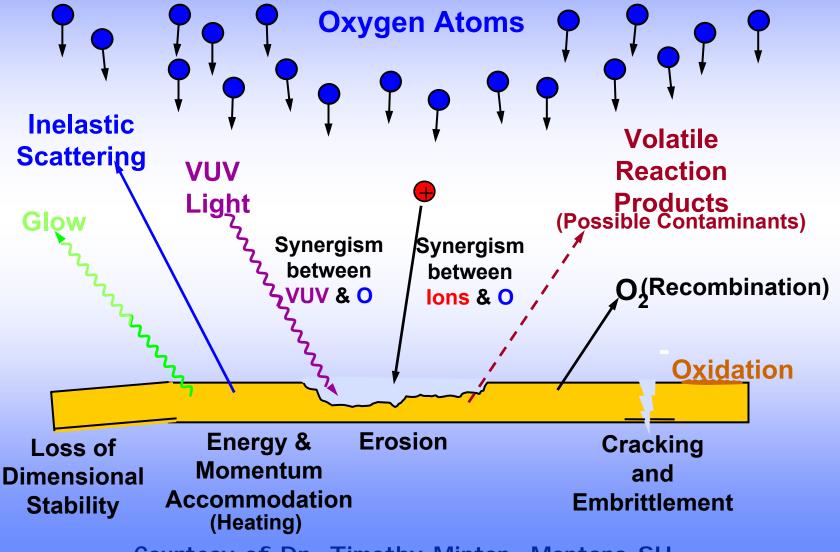
Lower Earth Orbit Environment

- ➤ Atomic Oxygen (AO) Erosion of Kapton in LEO is a serious threat to spacecraft durability.
- ➤ As a space vehicle orbits the Earth at orbital speed (7.8 km/sec at low altitudes) it undergoes energetic collisions with atoms and molecules in the orbital environment.
- ➤ AO is the dominant species in the outer ionosphere from 200-700 km, becoming as much as 90 % of the atmosphere at 500 km, a typical altitude for the International Space Station and future space platforms.



Atomic Oxygen and Synergistic Effects on Materials

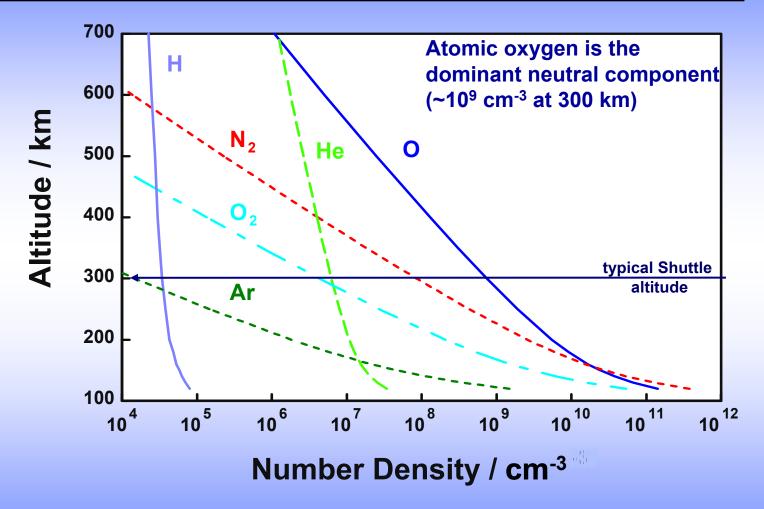




Courtesy of Dr. Timothy Minton, Montana SU



Neutral Composition of Earth's Upper Atmosphere



Roble, in <u>The Upper Mesosphere and Lower Thermosphere: A Review of Experiment and Theory</u>, Geophysical Monograph 87, pp 1 – 21, 1995.



Technical Problem Atomic Oxygen in Lower Earth Orbit



LEO Environment (Altitudes of 200 to 1500 km)

- Atomic Oxygen (AO): ~10⁶ 10⁸ atoms/cm³, up to 90 % of the atomosphere at 500km (typical altitude for international space station).
- Actual AO flux on spacecraft
 ~10¹² 10¹⁴ atoms/cm²•s
- AO Collision energy ~ 5eV
 (7.8 km/sec) (C-C bond energy ~ 4 eV, C-N ~ 3eV,
 Si-O ~ 8.3eV)
- Low-energy and high energy charged particles.
- Thermal cycling -50 to 150°C
- Solar VUV and UV radiation (~ 100 – 400 nm)
- Bond scission and radical formation can lead to embrittlement.

Bond	Dissociation Energy (EV)	λ (nm)	Material
-C ₆ H ₄ -C(=O)-	3.9	320	Kapton [®]
C-N	3.2	390	Kapton [®]
Si-O	8.3	150	Nanocomposite





State-of-the-Art Space Polyimides



- Kapton H Protected by a sputtered on Silica layer (SiO₂).
- Inherent problems in protective Silica layer:
- Defects from surface anomalies occurring during deposition process.
- Cracks and microdefects due to micrometeoroid and debris
- bombardment in LEO. Underpinning Effect.
- Results:
- Exposure of underlying Kapton layer.
- Lifetime of Kapton H protected by sputtered on silica layer:
- Example:
- Hubble Space Telescope:
- Altitude = 610 km.
- AO fluence exposure: 7.59 x 10²⁰ atoms/cm² for 3.6 years.
- Revisited every 2-3 years for maintenance including replacement of
- solar arrays and patching of multilayer insulation blankets.



AO undercutting of Aluminized-Kapton Multilayer Insulation flown on LDEF

Effect



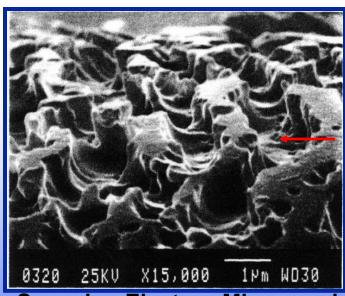
AO-Resistant

Cover or Coating

LDEF Satellite:

Long Duration Exposure Facility.

Total AO Exposure: 9 x 10²¹ atoms/cm² Depths of >0.0127cm (> 5mils) of Kapton sheets were eroded away after 5.8 yrs in LEO on the ram AO surface of the LDEF



Scanning Electron Micrograph Of Kapton MLI Surface.

Pinhole Caused by Defect Substrate Cavity Eroded by Multiple AO Entries **Underpinning** Nonreactive Collision Reactive Collision

Oxygen Atom

5 μm

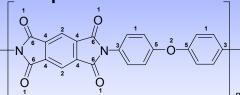


Goal



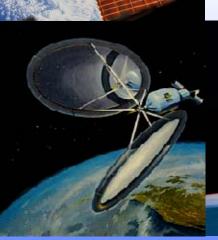






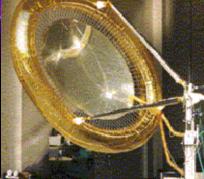
Our goal is to create an efficient drop-in replacement for Kapton that:

- Has increased space survivability due to resistance to atomic oxygen, thermal cycling, solar UV and VUV radiation, protons and electrons.
- 2. Is Self-Passivating and Self-Healing based on hybrid . organic/inorganic nanocomposite incorporation
- 3. Has superior optical properties, low solar absorptance, high thermal reflectance
- 4. Has excellent mechanical thermal properties.









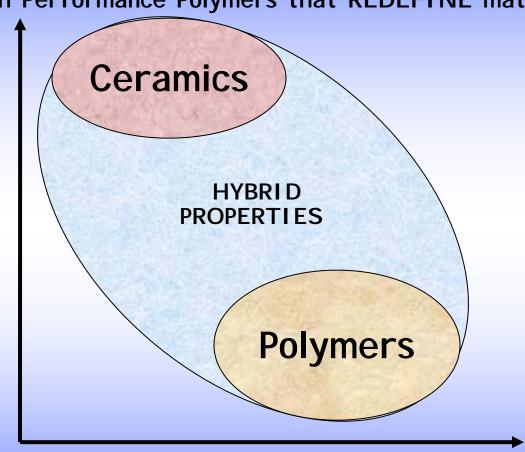


Hybrid Inorganic/Organic Polymers



Goal: Develop High Performance Polymers that REDEFINE material properties

Use Temperature & Oxidation Resistance



Toughness, Lightweight & Ease of Processing

Hybrid plastics bridge the differences between ceramics and polymers



Why Use POSS?



- ➤ Multifunctionality including no negative effects on processing (or can even get improvements)
- ➤ Properties previously not attainable (extended temp range, flame retardancy)
- ➤ Turnkey Utility
- > Control of molecular architecture

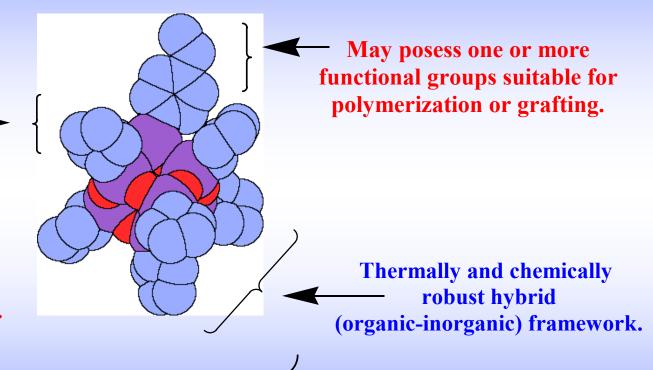


Anatomy of a POSS Nanostructure





Nanoscopic in size with an Si-Si distance of 0.5 nm and a R-R distance of 1.5 nm.



Precise three-dimensional structure for molecular level reinforcement of polymer segments and coils.



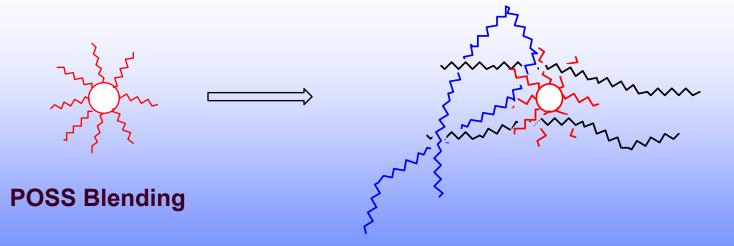
POSS Incorporation into Polymers



Cross-linker

Pendant Polymer

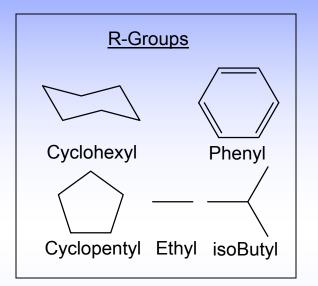
Bead Copolymer





New Polymer Feedstock Technology





Halides Nitriles Silanes Styryls

Alcohols Amines Silanols a-olefins

Esters I socyanates Silylchlorides Acrylics

Bisphenols Epoxides Norbornenyls

POSS-based macromers are available through either Gelest or Aldrich

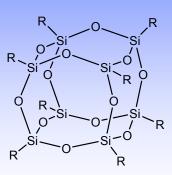
POSS technology is commercialized by Hybrid Plastics in Fountain Valley CA



POSS: Where We Were (1996)



- > Cost: \$5,000-\$10,000/lb
- Volume: ~20 lbs/yr
- Production time: min 11 days,
- > max 6 months
- Versatility: ~6 POSS feedstocks
- > ~30 POSS macromers
- No successful POSS blends
- ➤ Made only by U.S. Government





Why POSS and Why Nano?



1 mm	
	 Sewing Needle
	 Razor Blade Thickness
100 μm—	→ • Human Hair
	\
10 μm—	Most Cells & Fibers
·	
1 μm —	• Bacteria, Fillers &
	Polymer Morphology
100 nm	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \
10 nm —	• Viruses & Nanofillers
1.0 nm —	• POSS® Building Blocks - Macromolecules
0.1 nm —	🗦 • Atoms / Small Molecules

Field	Property	Critical Length	
Electronics	Tunneling	1-100 nm	
Optical	Quantum Well	1-100 nm	
	Wave Decay	10-1000 nm	
Polymers	Primary Structure	0.1-10 nm	
	Secondary Structure	10-1000 nm	
Mechanics	Dislocation Interaction	1-1000 nm	
	Crack Tip Radius	1-100 nm	
	Entanglement Rad.	10-50 nm	
Therm-Mech.	Chain Motion	0.5-50 nm	
Nucleation	Defect	0.1-10 nm	
	Critical Nucleus Size	1-10 nm	
	Surface Corrugation	1-10 nm	
Catalysis	Surface Topology	1-10 nm	
Biology	Cell Walls	1-100 nm	
Membranes	Porosity Control	0.1-5 nm	



Nanostructured™ POSS Chemicals Physical Form of Products





Hybrid Plastics

Crystalline Solids
Wide melting range 24°C to 400°C+

Waxes

Liquids & Oils

Wide viscosity range 40cSt. to 400cSt

>120 POSS Monomers, Polymers and Feedstocks Available



What Property Enhancements Can You Get From Using POSS?



increased T_g

increased T_{dec}

oxidation resistance

reduced flammability

extended use temperature range

altered mechanicals

reduced heat evolution

lower thermal conductivity

lower density



How to use POSS (Blends or Drop-In Nanofillers)

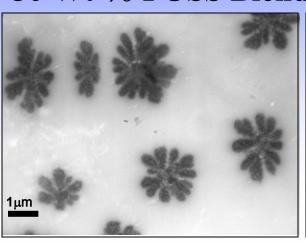


50 Wt % POSS Blends in 2 Million MW PS

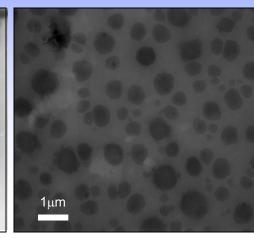


R = cyclopentyl

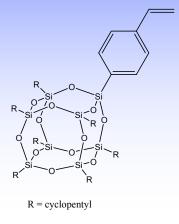
Cp₈T₈



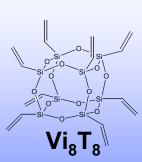
Domain Formation

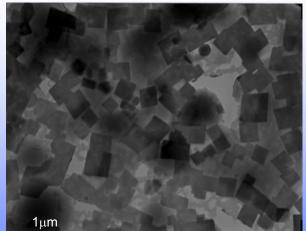


Partial Compatibility

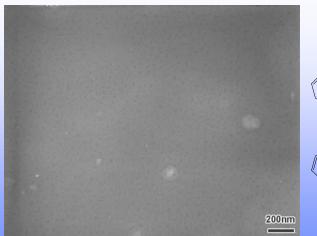


Cp₇T₈Styryl





Immiscible POSS Crystallites



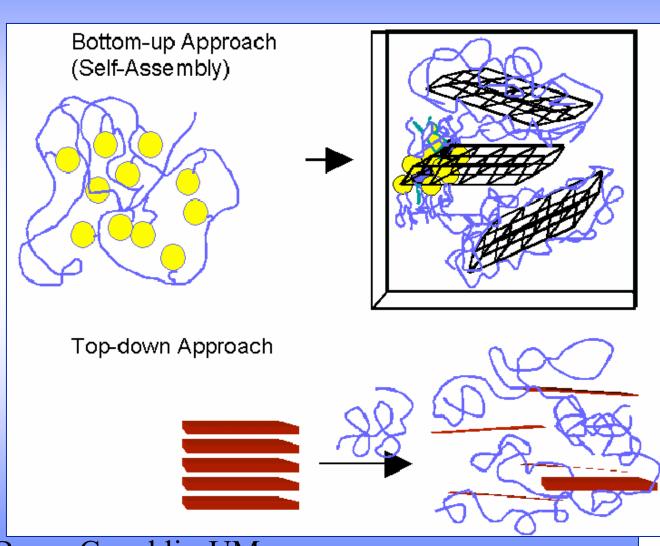
Phenethyl₈T₈

Complete CompatibilityPOSS Nanodispersion/Transparent



Coughlin Building Block Model (POSS Blends & Copolymers)









Bryan Coughlin-UMass



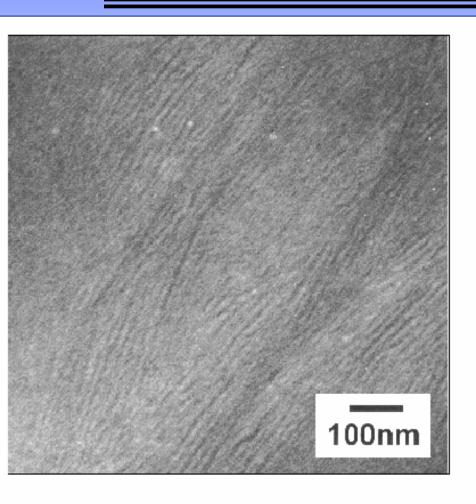
Coughlin Model Continued (building from the ground up)

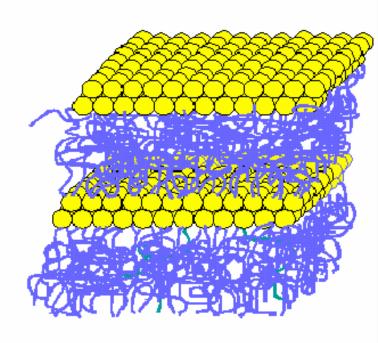




Nanoengineering with POSS







Bryan Coughlin-UMass

PBD-POSS4 (43wt%POSS)



Prof. Andre Lee i-PP/POSS Blends

e Si O Si O R O		N DD	'DD11 11				
Me Si O Si Me	Dow data	Neat <i>i</i> -PP (processed)	<i>i</i> -PP blended 2 wt%	<i>i</i> -PP blended 5 wt%	<i>i</i> -PP blended 10 wt%		
Me Me			Methyl ₈ T ₈	Methyl ₈ T ₈	Methyl ₈ T ₈		
Tensile	5000 psi	4800 psi	5000 psi	5100 psi	5200 psi		
Strength @	(34.5 MPa)	(33.0 MPa)	(34.5 MPa)	(35.1 MPa)	(35.8 MPa)		
Yield; ASTM	,		,				
D638							
Flexural	240 000	225 000:	251 000:	255 000	262 000		
Modulus	240,000 psi	235,000 psi	251,000 psi	255,000 psi	262,000 psi		
(0.05 in/min);	(1.655 GPa)	(1.620 GPa)	(1.730 GPa)	(1.757 GPa)	(1.80 GPa)		
ASTM D790A							
HDT @ 66 psi,	210 °F	210 °F	221 °F	239 °F	255 °F		
as injected;	(99 °C)	(99 °C)	(105 °C)	(115 °C)	(124 °C)		
ASTM D648	())	(5) (5)	(103 0)	(113 C)			
Impact Izod							
@25C	0.5 ft-lb/in	0.55 ft-lb/in	0.55 ft-lb/in	0.62 ft-lb/in	0.75 ft-lb/in		
ASTM D256A							

• The above data (other than Dow's data) is an average of at least 10 samples for each test with acceptable S.D. of 5% or better.

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Prof. A.

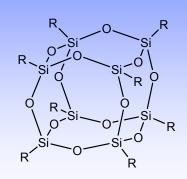
Prof. Andre Lee

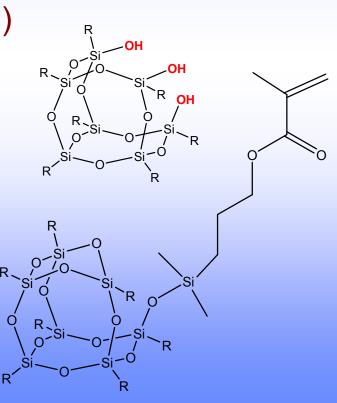


POSS: Where Are We Now (2004 1996 data in red

14) RODUCE RESERVAL LUBORITOR

- Cost: \$20-\$5000/lb (\$5000-\$1000/lb)
- Volume: Multi-ton (~20lb/yr)
- Production time: min 1 hour (11 days),
 max 14 days (6 months)
- Versatility: >120 POSS (36 POSS)
 monomers, feedstocks, polymers
- Many successful POSS blends
- Commercialized by Hybrid Plastics <u>www.hybridplastics.com</u>







Polyhedral Oligomeric Silsesquioxanes (POSS)



Hvbrid

RSiX₃ acid or base hydrolysis

Blendables

Resin

R Si O Si O Si O Si R O

Brown & Vogt:
JACS, 1965, 4313
Feher et al:
JACS, 1989, 1741;
Organometallics, 1991,
2526; Chem Comm,
1999, 1705, 2309

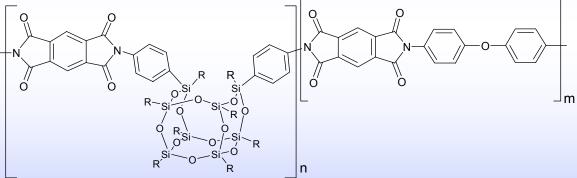


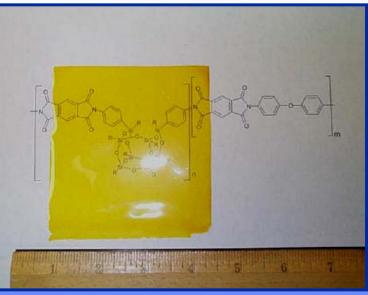
POSS-Kapton Polyimides



$$H_2N$$
 O NH_2







- transparent films
- no aggregates formed

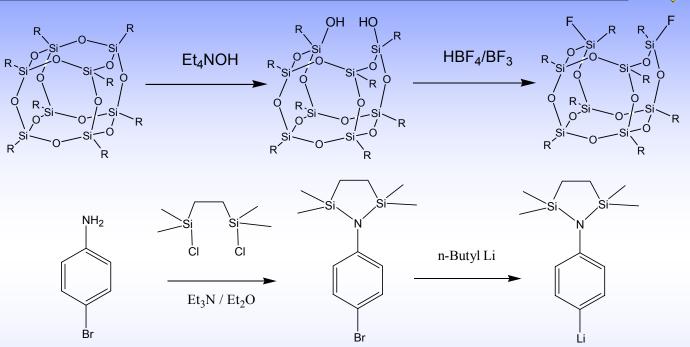


First POSS-Aniline Synthesis



Multiple step synthesis Moisture and air sensitive Not amenable to scale up

Yet Critical for Development of POSS Polyimides!!!





Program Challenge: Synthesis of Cost Efficient POSS Diamines.



Make a cost efficient POSS Polyimide that is amenable to scale up and performs as well as current POSS Polyimide (POSSdi1 Polyimide).

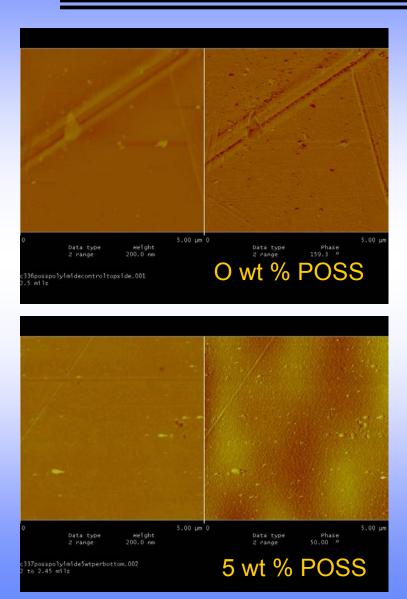
This will involve:

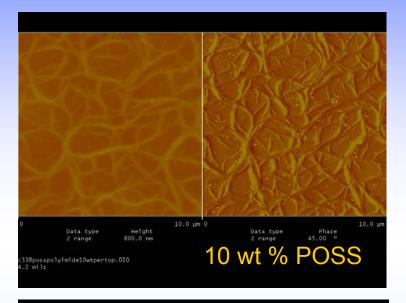
- ➤ Cost efficient synthesis of next generation POSS diamine monomers.
- ➤ Copolymerization of next generation POSS diamine monomers to form POSS Polyimides.
- >Testing of thermal and mechanical properties and range of optical clarity.
- ➤I maging of POSS Polyimide films: Atomic Force Microscopy (AFM), Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM) with X-Ray mapping.
- ➤ Molecular modeling and simulation of oxygen atom, photon, electron, and proton attack on next generation POSS Polyimides.
- ➤ Simulated LEO and GEO exposure ground based testing.

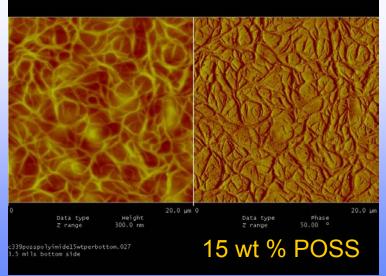
UMassAmherst



AFM I mages of Unexposed Polyimides Copolymerized With Various Weight Percents of POSS







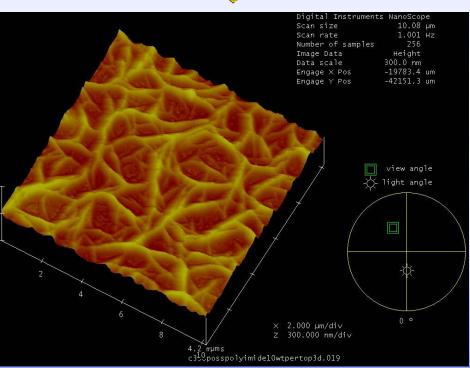


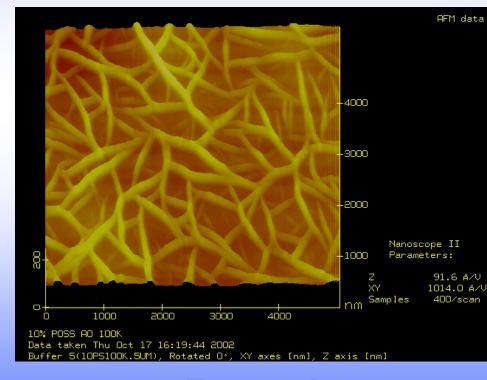
Three dimensional AFM Images 10 wt % POSS Polyimide Films











Unexposed

Exposed

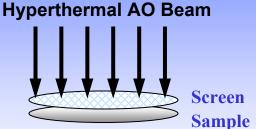


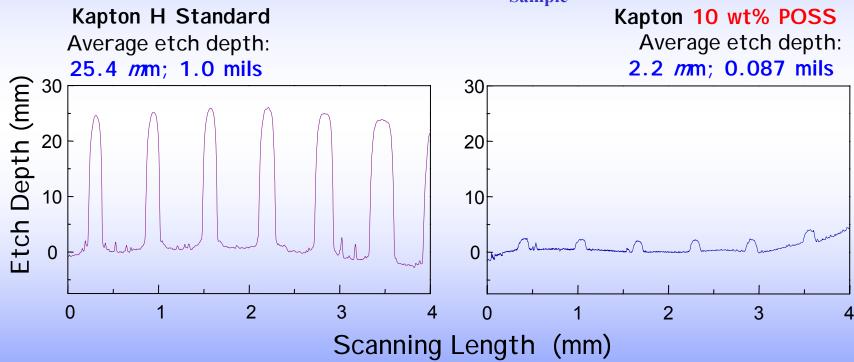
O-Atom Etching Experiment

Total AO fluence of 8.47×10^{20} atoms cm⁻² (100,000 pulses)









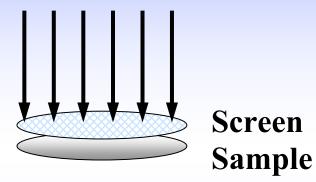
Significantly improved oxidation resistance due to a rapidly formed ceramic-like, passivating and **self-healing** silica layer preventing further degradation of underlying virgin polymer.



O-Atom etching experiment of POSS-Kapton polyimides Total AO fluence of 2.62 x 10²⁰ atoms/cm² (~ 3 Days in LEO)

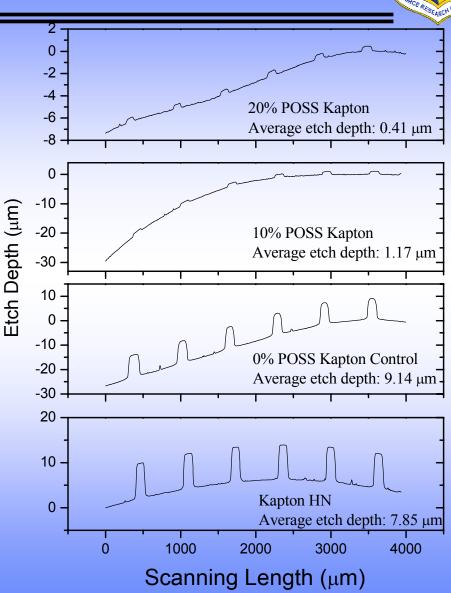


Hyperthermal AO Beam



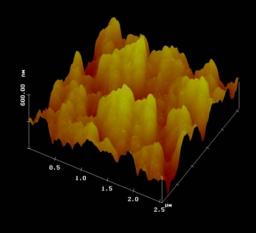
20 wt% POSS in Kapton results in over 20 time improvement in erosion resistance.

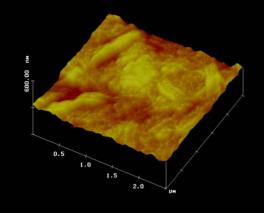


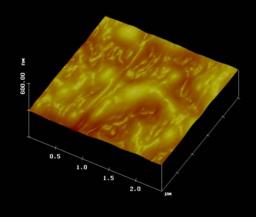




AFM A Falses lomeages exofost nexpensed in ROSS,000 Pulses of Housey theretal Firms AO Beam







00% POSS

rmscroughness: 1:1092 mm **10% POSS**

rms roughness: 1.0.3 nm 20% POSS

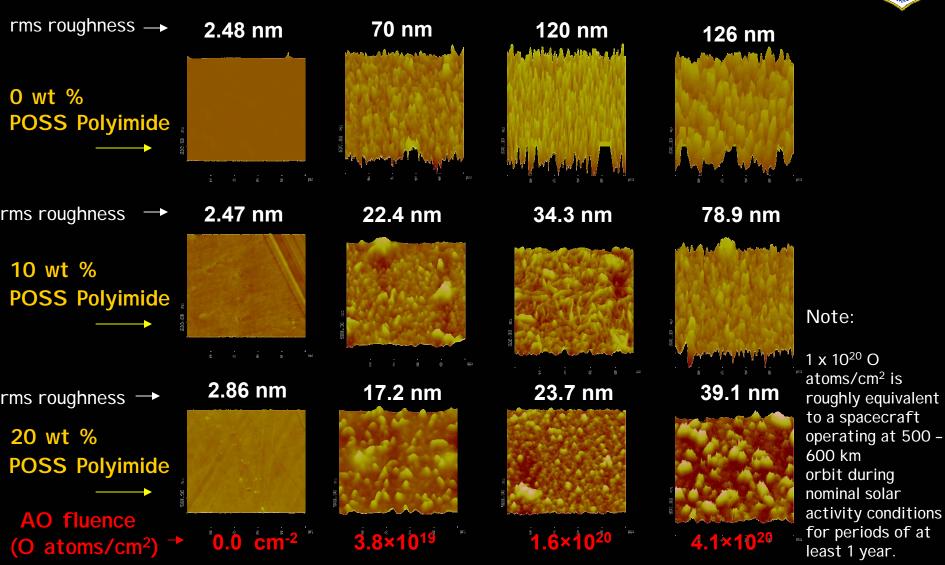
rmsercoughtnesse: 165755mm





AFM Images of POSS Polyimides With increasing AO Flux. $(10 \times 10 \mu m; z scale = 500 nm)$







X-ray Photoelectron Spectroscopy Analysis of POSS Polyimides



Surface Atomic Concentrations (%) determined from XPS Survey Scans following Atomic Oxygen Exposure

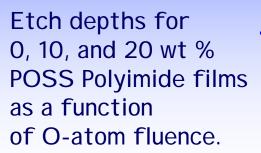
Sample	Exposure (beam pulses)	Kapton-equivalent atomic oxygen fluence (10 ²⁰ O atoms cm ⁻²)	С	Ο	Si	N
O wt% POSS polyimide	0 6 100 250	0 ~0.1 1.63 4.10	72 69 69 55	19.5 20 24 36	1 2 1 0	7 9 6 9
10 wt% POSS polyimide	0 6 100 250	0 ~0.1 1.63 4.10	77 73 48 20	16 18.5 30 56	2 5 19 23.5	5 3.5 3 0.5
20 wt% POSS polyimide	0 6 100 250	0 ~0.1 1.63 4.10	70 66 20 12	20 24 54 60	6 7 25 26	4 3 0 1



Erosion of POSS Polyimides by a Beam of Hyperthermal (5eV) O Atoms



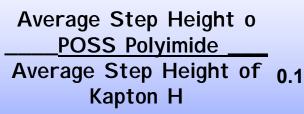
10

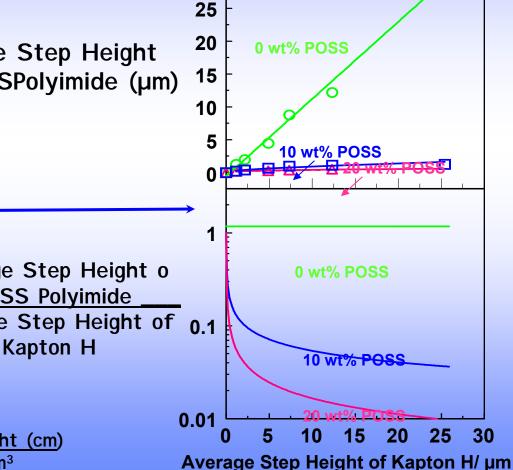


The erosion yields* of the 10 and 20 wt % POSS Polyimide samples were 3.7 and 0.98 percent, respectively, of the erosion yield for Kapton H at the highest fluence used in this experiment

(8.5x10²⁰ atoms cm⁻²).







30

Kapton-Equivalent Fluence / 10²⁰ O atoms cm⁻²

^{*}Erosion Yield = erosion depth step height (cm) AO fluence atoms/cm³



Molecular dynamics calculations of O(³P) collisions (5 eV) with POSS (Si₈O₁₂H₈) cages



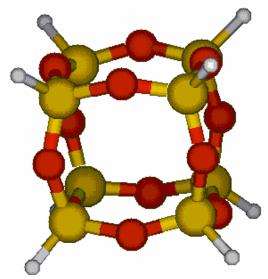
Possible Reaction Channels:

- ➤H abstraction to give OH
- ➤H elimination (O adds to the cage)
- **Cage opening (O adds to the cage)**

Number of trajectories	103		
Impact parameter	7 a.u.*		
Inelastic	63		
H abstraction	3		
H elimination	22		
Cage opening	15		

*(~Half diagonal of the Si₄ faces + 3 a.u.)



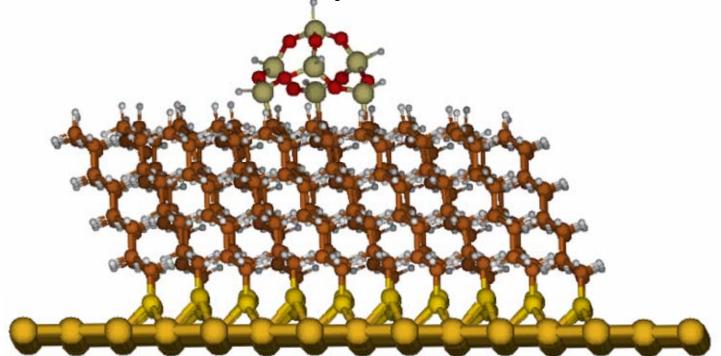




Model System: POSS Coated Alkane Thiol Self-Assembled Monolayer (SAM) on a Gold Surface.



- \triangleright Molecular dynamics calculations of $O(^{3}P)$ collisions (5 eV) with "functionalized" POSS.
- ➤ Method: Classical trajectories with a QM/MM (quantum mechanics / molecular mechanics) hybrid potential.
- QM part: O(3P), POSS cage and 1st methylene unit of the bound chains.
- MM part: All of the SAM but the 1st methylene unit of the bound chains.



➤ Results: Of limited trajectories studied, similar mechanisms of O atom attack on POSS (Si₈O₁₂H₈) Cages were found to apply to the studies of the POSS coated SAM.



Molecular Modeling Objectives



- 1. AO degradation pathways of polyimide when exposed to 5 eV oxygen.
- 2. Oxidation of POSS cage by 5 eV AO.
- 3. Penetration of 5 eV AO through POSS and silica layers to determine the SiO2 layer thickness needed to quench reaction with an underlying polymer layer.



MATERIALS INTERNATIONAL SPACE STATION EXPERIMENT

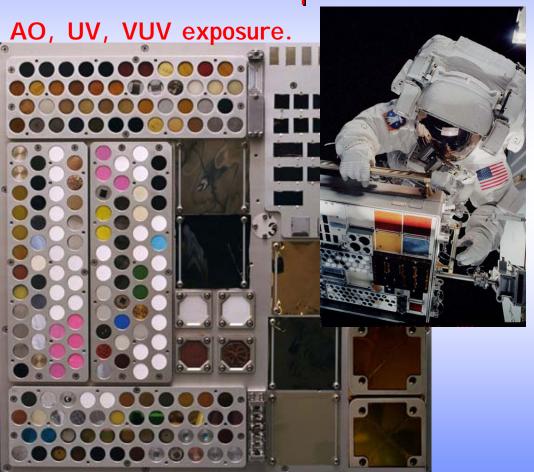




MATERIALS on the INTERNATIONAL SPACE STATION EXPERIMENT



POSS-Polymers Fly on STS 105 Discovery and are deployed on the International Space Station 16 Aug 2001, MI SSE 4



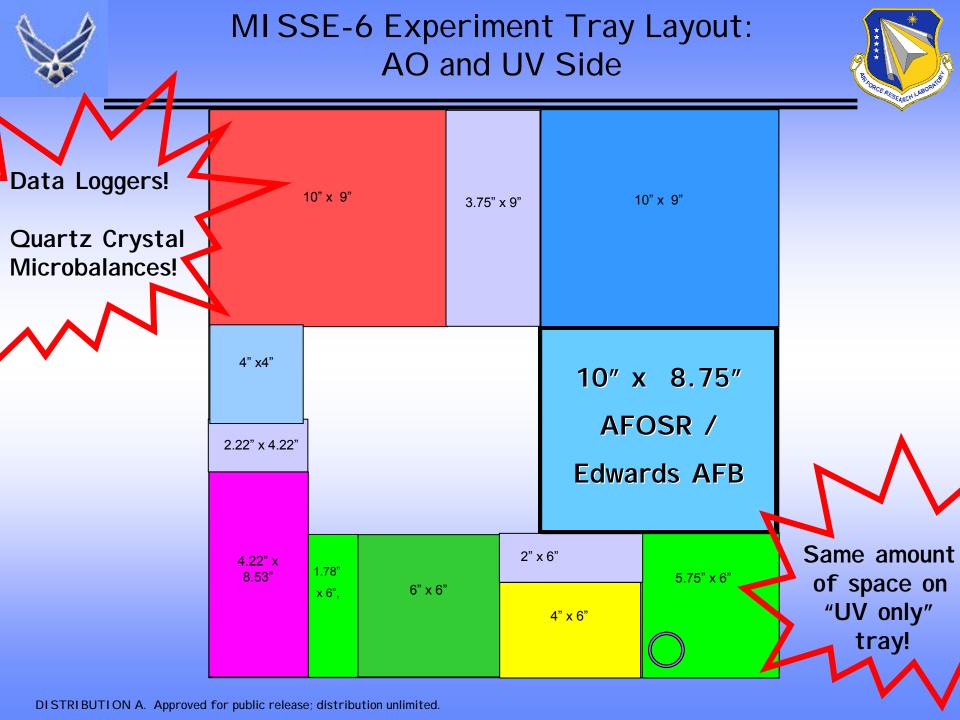
POSS-Polymers awaiting flight on ISS, MISSE 5

AO exposure only.

SALT Sprayed Samples!



Footage courtesy of NASA





Effects of POSS on Thermal and Mechanical Properties of Polyimides



POSS Polyimides do not lose rigidity above the Tg.

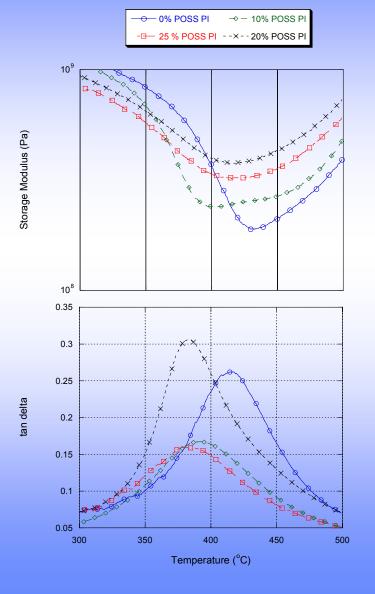
Room temperature modulus (stiffness) unaffected by POSS.

High temperature (430°C) modulus exhibits a maximum with POSS PI loading at 20 wt%:

@ 430°C, the modulus of20 % POSS Polyimide doublesrelative to 0 % POSS polyimide.

➤Tg of POSS polyimides is 5 - 10 % lower than polyimides (414°C).

Tan δ peaks for the 20 and 25 wt% are lower intensity and broader indicating that at POSS loadings greater than 20 wt% there exists interactions between the POSS molecules strong enough to significantly affect polymer chain dynamics.





Acknowledgments



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Hybrid Plastics

University of Montana State; Dr. Tim Minton Northwestern University: Dr. George Schatz



Summary and Conclusions



- Our goal is to create an efficient drop-in replacement for Kapton that is:
 - > Space survivable
 - > Self passivating
 - Self healing
 - > low in solar absorptance
 - Excellent in mechanical properties
- POSS Polyimides form a protective Si-O layer when exposed to Atomic Oxygen
- Modeling and Simulation Plan has shown that AO adds to the POSS molecule and does not pass through POSS.
- ➤ Thermal and Mechanical Testing indicate:
 - > POSS Polyimides do not lose rigidity above the Tg.
 - > Tg of POSS polyimides is 5 10 % lower than polyimides (414°C).
 - \succ Analysis of tan δ curves indicate that polymer chain dynamics are affected by the addition of POSS
- POSS-Polymers awaiting flight on ISS, MISSE 5 and scheduled to fly on MISSE 6.



Abstract



Polyimides such as Kapton are used extensively in spacecraft thermal blankets, solar concentrators, and space inflatable structures. Atomic oxygen (AO) in lower earth orbit (LEO) causes severe degradation in Kapton resulting in reduced spacecraft lifetimes. One solution is that SiO_2 coatings impart remarkable oxidation resistance and have been widely used to protect Kapton. Imperfections in the SiO_2 application process and micrometeoroid / debris impact in orbit damage the SiO_2 coating leading to erosion of Kapton.

A self passivating, self healing silica layer protecting underlying Kapton upon exposure to AO may result from the nanodispersion of silicon and oxygen within the polymer matrix. Polyhedral oligomeric silsesquioxane (POSS) composed of a inorganic cage structure with a 2:3 Si:O ratio surrounded by tailor able organic groups is a possible delivery system for nanodispersed silica. A POSS diamine was copolymerized with pyromellitic dianhydride and 4,4 '-oxydianiline resulting in POSS Kapton Polyimide. The glass transition temperature (Tg) of 5 to 20 weight % POSS Polyimide was determined to be 5 – 10 % lower than that of unmodified polyimides (414 °C). Furthermore the room temperature modulus of polyimide is unaffected by POSS, and the modulus at temperatures greater than the Tg of the polyimide is doubled by the incorporation of 20 wt % POSS.

To simulate LEO conditions, POSS Polyimide films were exposed to a hyperthermal O-atom beam. Surface analysis of exposed and unexposed films conducted with X-ray photoelectron spectroscopy, atomic force microscopy, and surface profilometry support the formation of a SiO₂ self healing passivation layer upon AO exposure. This is exemplified by erosion yields of 10 and 20 weight % POSS Polyimide samples which were 3.7 and o.98 percent, respectively, of the erosion yield for Kapton H at a fluence of 8.5 x 10²⁰ O atoms cm⁻².



POSS-Siloxane

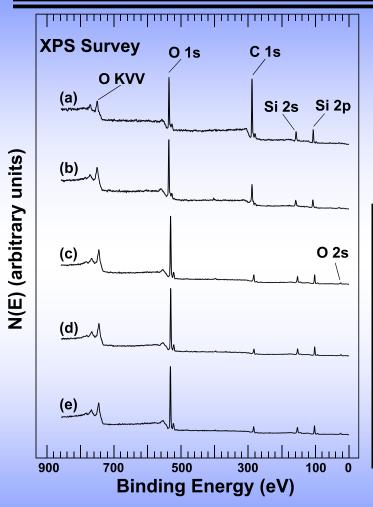


POSS-PDMS Copolymer



POSS Siloxane





Composition, at %

Sample Treatment	0	С	Si	
As entered	18.5	65.0	16.6	
2.0 hr	33.8	48.4	17.8	
24.6 hr	49.1	22.1	28.8	
63.0 hr	55.7	16.3	28.0	
4.8 hr air	52.8	19.5	27.7	

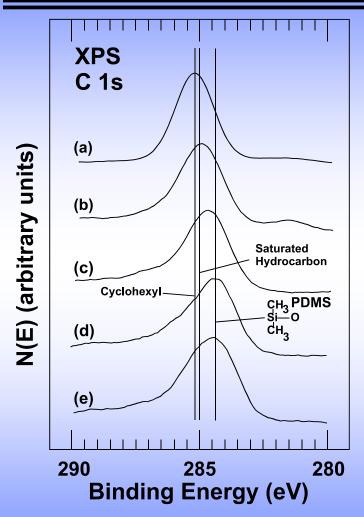
Gonzalez, R. I., Phillips, S. H., Hoflund, G. B., *J. of Spacecraft and Rockets*, Vol 37, No. 4, **2000**, pp. 463-467.

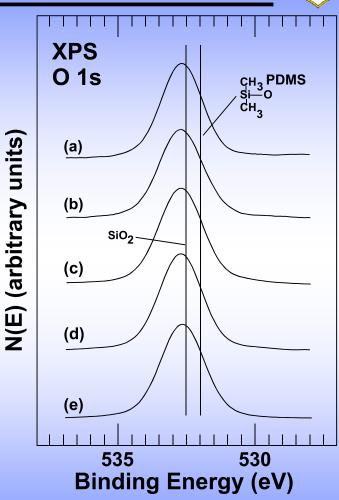
XPS survey spectra obtained from a solvent-cleaned, POSS-PDMS film (a) after insertion into the vacuum system, (b), after a 2-hr (c) 24.6-hr and (d) 63-hr exposure to the hyperthermal AO flux, and (e) 4.75-hr air exposure following the 63-hr AO exposure.



POSS PDMS





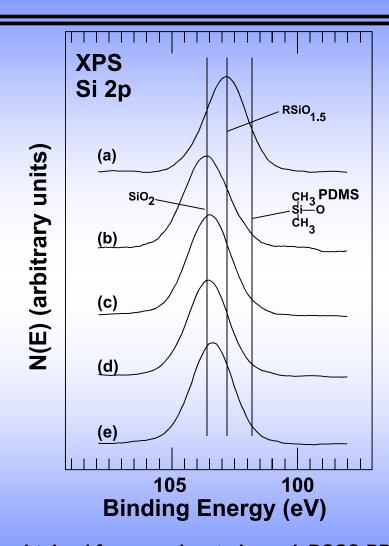


High Resolution C 1s and O 1s spectra obtained from a solvent-cleaned, POSS-PDMS film (a) after insertion into the vacuum system, (b), after a 2-hr (c) 24.6-hr and (d) 63-hr exposure to the hyperthermal AO flux, and (e) 4.75-hr air exposure following the 63-hr AO exposure.



POSS PDMS





High Resolution Si 2p spectra obtained from a solvent-cleaned, POSS-PDMS film (a) after insertion into the vacuum system, (b), after a 2-hr (c) 24.6-hr and (d) 63-hr exposure to the hyperthermal AO flux, and (e) 4.75-hr air exposure following the 63-hr AO exposure.



POSS-Polyurethane



R = cyclopentyl

POSS-TMP diol

Et₃N/DBTDL PTMG (M_n=2000) 1,4-butanediol

Fu, B.X., et al. *High Performance Polymers*, **2000**. 12(4): p. 565-571.

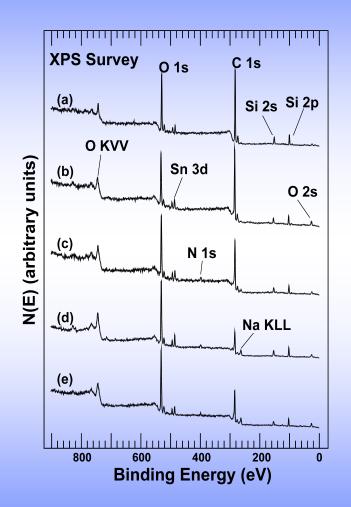
Fu, B.X., et al. *Polymer*, **2001**. 42(2): p. 599-611.

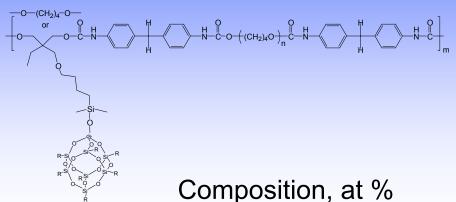
POSS



60 wt % POSS-Polyurethane







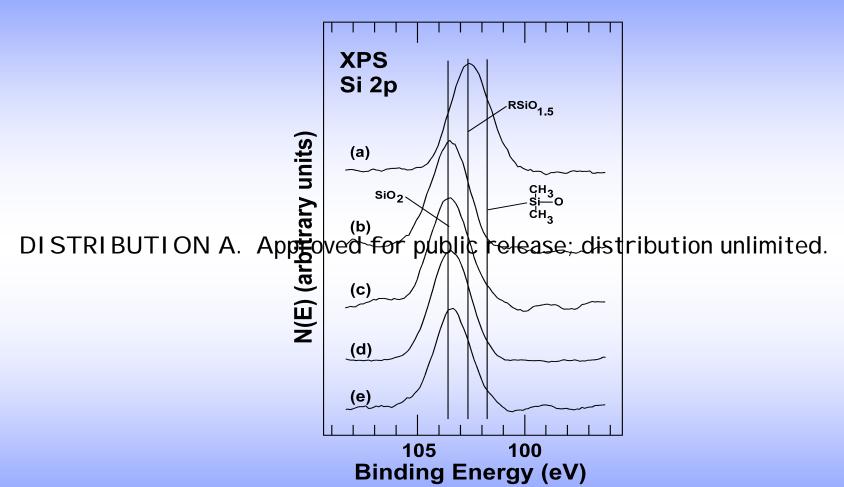
Sample Treatment	0	С	Si	Sn	Na	N
As entered	18.2	70.1	11.3	0.4	-	-
2.0-hr	17.5	70.2	11.2	0.7	0.4	-
24.0-hr	23.7	58.2	13.2	0.9	1.4	2.6
63.0-hr	35.3	37.3	20.4	1.3	3.0	2.7
3.3-h air	31.6	48.5	14.6	1.0	2.7	1.6

Phillips, S. H., Hoflund, G. B., Gonzalez, R. I., 45th International SAMPE Symposium, 2000, Vol. 45, No. 2, pp. 1921-1931.

DISTRIBUTION A. Approved for public release; distribution unlimited.







High Resolution Si 2p spectra from a 60 wt% POSS-PU (a) after insertion into the vacuum system, (b) after a 2-hr (c) 24-hr and (d) 63-hr exposure to the hyperthermal AO flux, and (e) 3.3-hr air exposure following the 63-hr exposure.



POSS High Performance Polyimides



POSS processable ether-imide

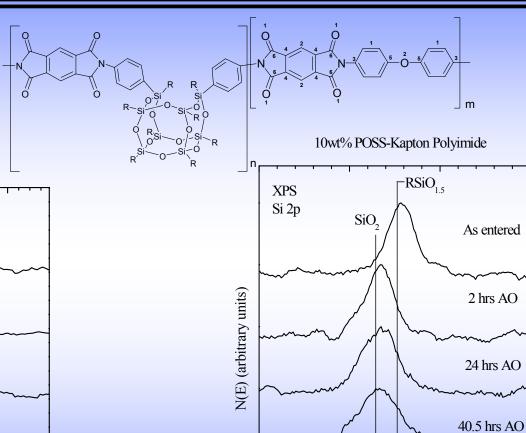
POSS-Fluorinated colorless polyimide



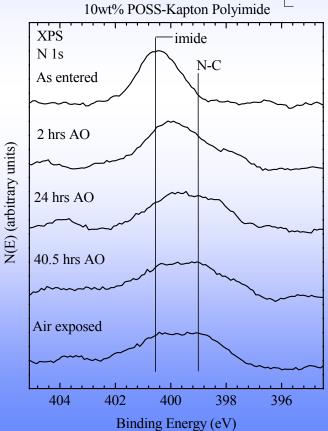
POSS Polyimide







110



Binding Energy (eV)

105

Air exposed

95

100